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(19)

(54) PROCESS FOR PREPARING A NYLON FILM

(71) We, FUJI PHOTO FILM CO., LTD., a Japanese Company, of No. 210, Nakanuma, Minami Ashigara-Machi, Ashigara-kamigun, Kanagawa, Japan, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process of preparing a nylon film having a fine pore structure.

Such films are mainly used to separate and remove fine grains and bacteria, contained in liquids and vapours.

In the past, such films have been manufactured by dissolving nylon in a suitable solvent, adding a gelling agent such as a lower aliphatic acid ester of a lower aliphatic alcohol, a lower aliphatic ether such as glycol, boric acid or citric acid and permitting the resulting liquid to flow over onto a flat plate, whereby a thin nylon film having a fine pore structure is obtained.

However, this method is not suitable for the manufacture of a bacteriological filter because not only is a long time required for the formation of a film due to the slow gelation, but also the pore size distribution is wide, e.g. 0.4 to 6.6 microns, due to unhomogeneous gelation.

The object of the present invention is to provide an improved process for manufacturing nylon films having a uniform, fine pore structure wherein the drawbacks of the prior art are eliminated.

Our process basically comprises dissolving nylon in an alcohol solvent, adding a cyclic ether as a gelling agent to the solution, permitting the resulting solution to flow over onto a flat support, and drying the film.

According to the process of the present invention, the time required for gelation is greatly shortened by up to one sixth; therefore, the fine porous film can be efficiently manufactured. In addition, the pore size distribution of films of the invention is remarkably uniform, e.g. from 0.7 to 1.3 microns.

As alcohol-soluble nylon resins used in the present invention, there may be mentioned, for example, CM 4000 and CM 8000 (products of Toray Inc.):

Composition	Molecular weight	Polymerization degree
CM 4000		
Copolymer of nylon 6 and nylon 66	20,000 to 30,000	100 to 150
CM 8000		
Copolymer of nylon 6, nylon 66 and nylon 610	20,000 to 30,000	100 to 150

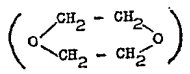
and Zytel 63 and Zytel 61 (products of du Pont Inc; Zytel is a Registered Trade Mark).

[*Price 25p*]

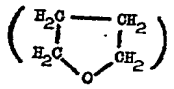
As solvents, there may be used, for example, methanol, ethanol, propanol or butanol.

As cyclic ethers which are used as the gelling agent in the present invention, there may be used, for example:

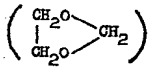
diethylene ether



tetramethylene oxide



or glycolmethylene ether



The preferred ratio by weight of ether: alcohol is 75:25 to 85:15.

The preferred ratio by weight of ether: nylon is 57:43 to 63:37.

Nylon films were prepared by the aforesaid method, under the following conditions, (a) with and (b) without a cyclic ether:

Alcohol solvent: methanol and ethanol
Cyclic ether: (a) diethylene ether and tetramethylene-oxide or (b) none
Nylon: copolymer of nylon 6 and nylon 66 and copolymer of nylon 6, nylon 66 and nylon 610.
Temperature at dissolution: 50° C to 75° C.
Casting temperature: less than 35° C.

The results obtained are shown in the following Table, in which the time for gelation represents the time required for the film to flow and to be whitened by gelation, and the pore size distribution represents the pore size range in which the pore size values obtained by measurement with a mercury intrusion porosity meter are in the range of from 10 to 90% in a cumulative distribution (see the accompanying drawing).

	(a) With Cyclic Ether	(a) Without Ether
Time for Gelation	15 — 20 minutes	1.5 — 2 hours
Pore Size Distribution	1.0 ± 0.3 micron (continuous line in drawing)	1.0 ± 0.6 micron (broken line in drawing)

From the results shown in the Table, it is seen that the process of the present invention gives a higher rate of gelation, a higher film-forming rate and a more uniform, fine pore size distribution as compared to the typical prior art process.

The present invention will now be illustrated by the following Examples. Parts are by weight.

EXAMPLE 1.

Sixteen parts of alcohol-soluble nylon CM 8000 as described above were dissolved in a solution consisting of 50 parts of methanol and 23 parts of water at a temperature of 50° to 75° C, and there were added thereto 10 parts of diethylene ether. The resulting nylon solution was caused to flow at a temperature below 35° C onto a support having a smooth surface and dried, whereby there was obtained a white porous nylon film of uniform, fine pore size distribution.

EXAMPLE 2.

Twenty parts of alcohol-soluble nylon CM 4000 were dissolved in a solution consisting of 60 parts of ethanol and 15 parts of water, and there were added thereto 14 parts of tetramethylene oxide. The resulting nylon solution was caused to flow on a smooth metal plate and dried, whereby there was obtained a white porous nylon film of uniform, fine pore size distribution.

WHAT WE CLAIM IS:—

1. A process for preparing a porous nylon film having a uniform pore size distribution, which comprises dissolving a nylon resin in an alcohol solvent, adding a cyclic ether to the solution, causing the resulting solution to flow onto a flat support, and drying the film thus produced.

2. A process according to claim 1, wherein the alcohol solvent is an aliphatic alcohol of 1 to 4 carbon atoms.

3. A process according to claim 1 or 2,

wherein the cyclic ether is diethylene ether, tetramethylene oxide or glycolmethylene ether.

4. A process according to claim 1, 2 or 3, wherein the ratio by weight of ether to alcohol is 75:25 to 85:15.

5. A process according to any preceding claim, wherein the ratio by weight of ether to nylon resin is 57:43 to 63:37.

6. A process for preparing a porous nylon film, substantially as hereinbefore described in either of the foregoing Examples.

7. Porous nylon films made by a process as claimed in any preceding claim.

8. Films as claimed in claim 7 having pores of a size of 0.7 to 1.3 microns.

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1345349 COMPLETE SPECIFICATION

1 SHEET *This drawing is a reproduction of the Original on a reduced scale*